Materials Letters 154 (2015) 17–20

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

Tailoring the pore morphology of porous nitinol with suitable mechanical properties for biomedical applications

Irfan Haider Abidi, Fazal Ahmad Khalid, Muhammad Umer Farooq*, Muhammad Asif Hussain, Adnan Maqbool

Faculty of Materials Science and Engineering, GIK Institute of Engineering Sciences and Technology, Topi 23640, Sawabi, Khyber Pakhtunkhwa, Pakistan

ARTICLE INFO

Article history:
Received 12 November 2014
Accepted 11 April 2015
Available online 20 April 2015

Keywords:
NiTi
Shape memory materials
Superelasticity
Biomaterials
Porous materials

ABSTRACT

NiTi, commercially known as Nitinol, exhibits unique properties of high recoverable strain, shape memory effect, biocompatibility, high strength, low density and stiffness. The porous structure of this material mimics the hierarchical structure of bone, which makes it a potential material for biomedical implants. Porous NiTi shape memory alloys were fabricated by pressureless sintering under vacuum using NaCl as a spacer. The porosity of the fabricated alloys was kept within the range suitable for better bone tissue ingrowth and osseointegration for bone grafting and implant applications. The effect of the shape and size of NaCl powder on the pore morphology and mechanical properties was studied. Pore morphology and size were replica of the shape of the spacer powder. Moreover, porous alloys with spherical pores showed better mechanical properties as compared to the cuboidal shape. All the foams showed low modulus and almost complete recovery at 2% strain, which is critical to mimic the bone structure for implant applications.

1. Introduction

NiTi shape memory alloys, also known as Nitinol, have gained technical importance equally in biomedical and aerospace industries due to their excellent mechanical and shape memory properties [1,2]. Especially for biomedical implants, the properties such as low modulus comparable to bones, adequate yield strength, higher ductility and elasticity, good corrosion resistance and biocompatibility are essential for long-term performance [3,4]. As compared to other Ti-based shape memory alloys [5,6] and metals e.g. SS316, Co–Cr alloys, Nitinol proves to be the most suitable material for biomedical implants due to its low modulus and better biocompatibility [7,8]. Moreover, recently porous NiTi has enhanced its popularity as an implant material, as it provides a unique cellular structure which facilitates ingrowth of tissues, medicament transportation, exchange of nutrition and lower stiffness for better integration with the host bone [9]. In addition, superelastic property of porous Nitinol mimics the reversibility of human bone, which can recover strains up to 2% [10]. The porous NiTi has been fabricated through various powder metallurgy techniques [11–13]. In this study, we fabricated porous NiTi by melting off sodium chloride spacer powder through conventional sintering.

2. Experimental

Porous NiTi was fabricated by using sodium chloride powder (N400-60% cuboidal and N400-60% spherical morphology) as spacer during sintering process to check the effect of shape of spacer. Three types of NaCl powders with mean particle size of 265 μm, 425 μm and 600 μm were used to synthesize samples coded as N200-50%, N400-50% and N600-50% respectively, to investigate the effect of size variation of the spacer. The Ni (≤ 2 μm) and Ti (≤ 63 μm) powders were mixed with 50.5 at% Ni—49.5 at% Ti composition in planetary ball mill at 200 rpm for 3 h. The NaCl powders were blended with premixed powders in 50–60 Vol% in rotating-cylindrical mixer for 12 h. The powders were compacted with aspect ratio of approximately one by uniaxial pressing at 300 MPa. The samples were sintered under high vacuum $< 10^{-5}$ mbar at 1000 °C for 4 h, maintaining heating and cooling rate of 7 °C/min. Desalination in circulating water was carried out overnight to ensure complete removal of NaCl from the foam. Philips X-Ray diffractometer was used to investigate phase constituents. Different phases present were studied by etching the polished surfaces with Kroll's reagent ($10HNO_3$–$2HF$–$88H_2O$).

* Corresponding author. Tel.: +92 3216705295.
E-mail address: umer_534@yahoo.com (M.U. Farooq).

http://dx.doi.org/10.1016/j.matlet.2015.04.057
0167-577X/

Instron-8501 100 kN testing machine was used for uniaxial compression testing, using a strain rate of $1 \times 10^{-4}\,s^{-1}$.

3. Results and discussion

The porous NiTi samples with cuboidal and spherical shape pores are shown in Fig. 1(a) and (b). The almost complete removal of NaCl from samples by sintering above the melting point of salt and desalination was confirmed by the absence of Na or Cl peaks in EDS spectrum in Fig. 1(c). Average pore size was calculated for various samples with different pore size using a line intercept method [14]. The average porosity of porous samples was 63.6% and 53.4% for 60 Vol% and 50 Vol% NaCl spacer, respectively, calculated by Archimedes principle. The porosity was within the range necessary for the implants i.e. 30–90% for better bone tissue ingrowth and integration [12].

The marginally higher porosity than the NaCl amount was accounted for the microporosity, that can be observed within the matrix in micrographs Fig. 1(a), (b) and (e). Two kinds of porosity were clearly visible in all micrographs. Large pores were present due to the spacer powder replication and small pores resulted from microporosity, which is often present in pressureless sintering techniques [15,16]. Fig. 1(a) and (b) reveals the fact that pore shape and size were replicated (a) cuboidal and (b) spherical NaCl handsomely. The interconnectivity between pores has been indicated by arrows. The interconnected pores are beneficial for the tissue ingrowth [3].

The XRD pattern of the Ni and Ti powders and porous NiTi samples after sintering are shown in Fig. 1(d). Since the XRD

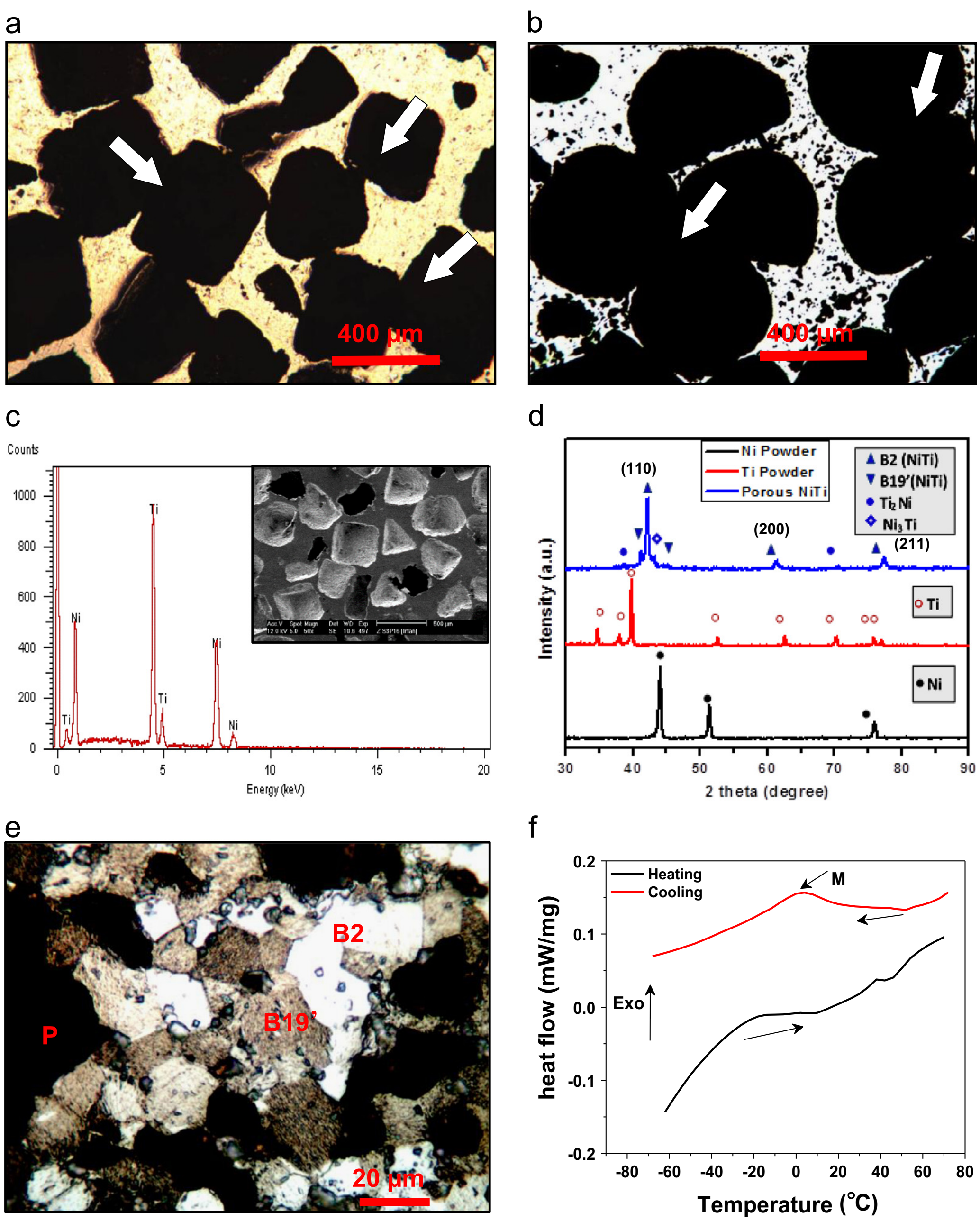

Fig. 1. Optical micrographs of porous NiTi fabricated with replication of (a) 60% NaCl cuboidal (b) 60% NaCl with spherical shape powder. Arrows pointing out the interconnectivity present. (c) EDX analysis of porous NiTi. Inset is the SEM micrograph of foam (d) XRD Spectra of porous NiTi and elemental Ni and Ti powders (e) Optical micrograph of porous NiTi after etching and (f) DSC analysis of porous NiTi showing transformation temperatures.